



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7 : C08L 91/08, 91/06, C11C 5/00	A1	(11) International Publication Number: WO 00/60012 (43) International Publication Date: 12 October 2000 (12.10.00)
(21) International Application Number: PCT/EP00/02848 (22) International Filing Date: 30 March 2000 (30.03.00) (30) Priority Data: 99302573.3 31 March 1999 (31.03.99) EP (71) Applicant: SHELL INTERNATIONALE RESEARCH MAATSCHAPPIJ B.V. [NL/NL]; Carel van Bylandtlaan 30, NL-2596 HR The Hague (NL). (72) Inventors: MACE, Jean-Michel; Avenue Jean Monnet 1, B-1348 Ottignies, Louvain-La-Neuve (BE). ROUMACHE, Olivier; Avenue Jean Monnet 1, B-1348 Ottignies, Lou- vain-La-Neuve (BE).		(81) Designated States: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: WAX COMPOSITIONS COMPRISING WAX AND OIL (57) Abstract The present invention provides a method for reducing bleeding number in wax compositions comprising wax and oil, wherein said waxes are selected from petroleum and synthetic waxes and said oil is present in the range of 1 to 45 % w/w, by incorporating in the composition an effective amount of an oil retention agent in the form of an elastomeric polymer. The invention has particularly useful application to wax compositions used for wax candles. The invention further provides the use of elastomeric polymers for reducing bleeding number in wax compositions.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

- 1 -

WAX COMPOSITIONS COMPRISING WAX AND OIL

Field of the Invention

The present invention relates to wax compositions comprising wax and oil.

Background of the Invention

5 Fully refined petroleum waxes have numerous industrial applications including use in adhesives, candlemaking, food coatings, lubricants, paper coatings and waterproofing.

10 In a typical sequence of wax production, long residue is subjected to vacuum distillation, leading to spindle oil distillate, light machine oil distillate, medium machine oil distillate and distillation residue as typical products. Such products may be subjected to solvent refining and de-waxing to yield wax products such
15 as spindle oil slack wax (SPO wax), light machine oil slack wax (LMO wax), medium machine oil slack wax (MMO wax) and bright stock slack wax (BSO) wax. These waxes may be treated by re-pulping processes, typically leading to waxes having >3% w/w oil content, or solvent
20 de-oiling, typically leading to waxes having <1% w/w oil content.

The initial de-waxing stage removes a relatively small proportion of wax from a large proportion of oil. This operation is usually performed by 'solvent de-
25 waxing' in which the waxy feed is mixed with a blend of solvents, chilled to crystallise the wax, and then filtered to remove the wax. At this stage SPO wax, LMO wax, MMO wax and BSO wax fractions usually contain between 10 to 35% w/w oil.

30 The presence of oil in a petroleum wax has a negative influence on its final properties. As the oil content

- 2 -

increases, the tensile strength, hardness and resistance to scuffing are impaired. The oil content of such waxes may cause staining upon contact with paper.

5 In candlemaking, for example, the presence of excess oil can result in both the candles bending and dripping during use at ambient temperature and also tacking/sticking during storage at room temperature.

10 In order to mitigate the negative effects that oil impart on the wax, de-waxing is usually followed by an expensive de-oiling step to reduce the oil contents to typical levels of <1% w/w. This is usually achieved by a 'solvent de-oiling' process. Solvent de-oiling is essentially similar to solvent de-waxing but is designed to remove a relatively small amount of oil from a larger
15 proportion of wax. The operation is controlled to produce a wax of the required oil content and melting point. An older 'sweating' process can be used to de-oil coarsely crystalline paraffin waxes. The sweating process results in a crude fractionation of the wax components wherein
20 lower melting point waxes are removed along with the oil.

The Foots oil (approximately 12-20% w/w) that results from such de-oiling processes is normally sent to a catcracker. Hence solvent de-oiling is both a costly and wasteful process.

25 The tendency of a wax to 'bleed' or sweat oil can be evaluated by determining its bleeding number. Tests for assessing the bleeding number of petroleum waxes are known in the art and are described, for example, in *Petroleum Refiner*, 1948, Vol. 27, No. 8, pp. 429-431.

30 It is highly desirable to be able to produce waxes that do not 'bleed' or sweat oil, without having to employ the full de-oiling process.

Summary of the Invention

35 The present invention provides a method for reducing bleeding number in wax compositions comprising wax and

- 3 -

oil, wherein said waxes are selected from petroleum and synthetic waxes and said oil is present in the range of 1 to 45% w/w, by incorporating in the composition an effective amount of an oil retention agent in the form of an elastomeric polymer.

The invention has particularly useful application to wax compositions used for wax candles.

The invention further provides the use of elastomeric polymers for reducing bleeding number in wax compositions.

Detailed Description of the Invention

The present invention provides a method for reducing bleeding number in petroleum and synthetic wax compositions containing wax and oil, wherein an oil retention agent in the form of an elastomeric polymer is incorporated in the composition. The incorporation of elastomeric polymer makes it feasible to avoid having to fully de-oil the wax prior to use, depending upon the bleeding tendency required in the composition.

Examples of waxes that can be treated by the invention are paraffin waxes and paraffin wax blends, preferably SPO, LMO, MMO and BSO waxes.

The process can be equally applied to synthetic waxes, such as Fischer-Tropsch (FT) waxes. Said FT waxes are hydrocarbon waxes that are produced by the reaction of carbon monoxide and hydrogen in the presence of a catalyst.

Petroleum and synthetic waxes that may be conveniently treated by the present invention preferably have an oil content in the range of from 1 to 35% w/w and most preferably in the range of from 1.5 to 15% w/w.

Elastomeric polymers are generally associated with polymers of conjugated dienes, such as butadiene or isoprene, or with copolymers of conjugated dienes with another copolymerisable monomer for example a mono vinyl

- 4 -

aromatic hydrocarbon, such as styrene. It is emphasised that the elastomeric polymer used in the present invention is not restricted to such polymers or copolymers and may include any polymer with elastomeric (i.e. rubbery) properties. Suitable elastomeric polymers include elastomeric polymers of olefins, diolefins and cyclic olefins amongst others those that have been produced using metallocene catalysts (metallocene polymers). However, the polymers of conjugated dienes, or copolymers of dienes are the preferred elastomeric polymers in respect of this invention. These polymers may be random and/or block copolymers.

The preferred elastomeric polymers in respect of this invention are block copolymers of at least one mono vinyl aromatic monomer and at least one conjugated diene. More preferably, the block copolymer contains at least one predominantly poly(mono vinyl aromatic) block and at least one predominantly poly(conjugated) diene block. Optionally, the poly(conjugated diene) block may be completely, partially or selectively hydrogenated.

With the term "predominantly" is meant that the main monomer of the respective individual blocks optionally may be mixed with minor amounts (e.g. amounts less than 50% mol/mol) of another comonomer and more in particular with minor amounts of the main monomer of other blocks.

Examples of the mono vinyl aromatic monomers may be selected from styrene, α -methylstyrene, p-methylstyrene, m-methylstyrene, o-methylstyrene, p-tert-butylstyrene, dimethylstyrene, and various other alkyl-substituted styrenes, alkoxy-substituted styrenes vinyl naphthalene and vinyl xylene. The alkyl and alkoxy groups of the alkyl-substituted or alkoxy substituted styrenes respectively preferably contain from 1 to 6 carbon atoms, preferably from 1 to 4 carbon atoms. The conjugated diene monomers are preferably conjugated dienes with from 4 to

- 5 -

8 carbon atoms per monomer, for example, butadiene, isoprene, 2-ethyl-1,3-butadiene, 2,3-dimethyl-1,3-butadiene, 1,3-butadiene, 1,3-pentadiene, 2,4-hexadiene, 3-ethyl-1,3-pentadiene, and mixtures thereof.

5 Of these monomers styrene is the preferred vinyl aromatic monomer. Butadiene or isoprene or mixtures thereof are the preferred conjugated dienes. Block copolymers which contain only substantially pure poly(butadiene) or pure poly(isoprene) blocks are
10 particularly preferred.

 The apparent molecular weight of the elastomeric polymer may conveniently be in the range of from 20,000 to 750,000 and preferably in the range of from 40,000 to 730,000 and more preferably in the range of from 60,000
15 to 700,000.

 With the term "apparent molecular weight" as used throughout the specification is meant the molecular weight of a polymer, as measured with gel permeation chromatography (GPC) using poly(styrene) calibration
20 standards (according to ASTM D 3536).

 The elastomeric block copolymers may be linear triblock or multiblock copolymers or multi-armed or star shaped symmetrical or unsymmetrical block copolymers. Diblock copolymers may also be used, as well as mixtures
25 of block copolymers such as triblock copolymers containing diblock copolymers.

 The elastomeric block copolymers, which are incorporated in wax compositions described herein, may be prepared by any method known in the art including the
30 well known full sequential polymerisation method, optionally in combination with reinitiation, and a coupling method, as illustrated in e.g. US-A-3,231,635; US-A-3,251,905; US-A-3,390,207; US-A-3,598,887; US-A-4,219,627; EP-A-0,413,294; EP-A-0,387,671;
35 EP-A-0,636,654, and WO 04/22931. Examples of coupling

- 6 -

agents used in this coupling method are, for example, dibromoethane, silicon tetrachloride, diethyl adipate, divinylbenzene, dimethyldichlorosilane, methyldichlorosilane. Particularly preferred in such a preparation route is the use of non-halogen containing coupling agents, for example gamma-glycidoxypropyltrimethoxysilane, and 'EPON' 825 (TM, diglycidylether of bisphenol A).

The vinyl aromatic monomer content of the elastomeric polymer is preferably in the range of from 0 to 70% w/w, more preferably from 0 to 50% w/w.

As indicated above, the elastomeric polymer may and indeed preferably is a hydrogenated block copolymer.

The elastomeric polymer may be hydrogenated by any suitable technique. The hydrogenation may be complete or partial. In addition, it is possible to selectively hydrogenate just the non-aromatic (i.e. ethylenic) unsaturation so as to convert, e.g. a polyisoprene block to an ethylene propylene rubber (EPR) block.

The poly(conjugated diene) blocks can be hydrogenated up to a substantial degree, i.e. more than 80% of the original ethylenic unsaturation has been hydrogenated. According to the preferred embodiment of the present invention, the poly(conjugated diene) blocks have been hydrogenated up to a residual ethylenic unsaturation (RU) of at most 10%, and most preferably less than 5%.

Examples of elastomeric polymers which can be conveniently used in the present invention include elastomeric block copolymers sold under the trademarks 'KRATON' D and G polymers (TM, SHELL Group of Companies), 'VECTOR' (TM, DEXCO), 'EUROPRENE' (TM, ENICHEM), 'QUINTAC' (TM, NIPPON ZEON), 'FINAPRENE' (TM, FINA), 'STEREON' (TM, FIRESTONE), 'TUFFPRENE' (TM, ASAHI), and 'SEPTON' (TM, KURARAY) and more preferably 'KRATON' elastomeric block polymers sold under the trade

- 7 -

designations of 'KRATON' D1102, G1650, G1652, G1654, G1657, G1726, G1750 and G1780 are used. Suitable metallocene polymers that may be used in the course of the present invention include those sold under the trade designations 'ENGAGE 8200' (DOW) and 'EXACT 4049' (EXXON).

It will be appreciated that mixtures of elastomeric polymers may also be incorporated in the wax composition.

For cost reasons, the amount of elastomeric polymer incorporated in the wax composition in order to reduce bleeding number will generally be kept as low as possible. The amount of elastomeric polymer incorporated in the wax composition is preferably in the range of from 0.1 to 10% w/w, and more preferably in the range of from 0.1 to 5% w/w based on wax composition.

The incorporation of elastomeric polymer in the wax composition is preferably in an amount such as to reduce the bleeding number of the wax composition by at least 10%, preferably at least 30% and more preferably at least 50%, measured at a temperature of generally at least 30 °C, preferably at least 35 °C, more preferably at least 40 °C and most preferably at least 45 °C. Most preferably, the elastomeric polymer is incorporated in an amount sufficient to reduce the bleeding number of the wax composition to generally less than 8 mm, preferably less than 6 mm, more preferably less than 2 mm and most preferably 0 mm, measured at a temperature of generally at least 30 °C, preferably at least 35 °C, more preferably at least 40 °C and most preferably at least 45 °C. Moreover, the incorporation of elastomeric polymers in wax compositions can significantly increase the hardness of the waxes produced.

Incorporation of the elastomeric polymer in the wax composition is preferably done in a manner which achieves a homogeneous or substantially homogeneous incorporation

- 8 -

of the polymer into the wax. This may conveniently be achieved by physical blending.

Blending of elastomeric polymers into wax compositions requires the combination of a suitable degree of shear at a suitable temperature. As the amount of elastomeric polymer present in the composition will be low, the temperature is the most significant parameter. But, to reduce the blending time it is necessary to apply some mechanical shear. Thus, temperature is the more significant parameter and little or no mechanical shear may be necessary.

This minimum operating temperature is strongly related to the molecular weight of the wax and that of the elastomeric polymer; in particular that of the polystyrene end-block when a polystyrene-containing block copolymer is used. The temperature will usually be the maximum temperature the wax can withstand with a maximum of 250 °C. As an example, a 30 °C wax, that is to say a wax with a melting point of 30 °C, will require 120 °C with a low molecular weight elastomeric polymer and 190 °C with a high molecular weight elastomeric polymer.

The use of a high shear mixer makes it possible to work at temperature slightly lower than usually needed for a conventional mixer.

For instance, the modification of the wax may be performed with a high shear rotor/stator mixer equipment. Many other techniques may also be employed to provide a homogeneous wax/elastomeric polymer composition, including but not limited to the use of internal mixer, Z-blade and extruder (screw has to be designed for super low viscosities) equipment.

Wax compositions described in the present invention can be conveniently used in a wide range of applications. The present invention can be suitably employed in traditional wax applications wherein oil retention is an

- 9 -

important feature of use. Use of wax compositions provided by the present invention includes candles, adhesives, dipping, carbon papers, crayons, dental modelling, food coating, matches, packaging material, polishes, electrical specialities, metal casting, moisture resistance, binders and metal injection moulding.

The wax composition can be further combined with other additives including but not limited to stearic acid, mould releases, colourants, dyes, odourants, UV inhibitors, modifiers and hardening resins.

In a preferred embodiment of the invention said compositions are employed in the production of wax candles.

Candles are typically made by casting, compression, dipping, drawing, extrusion, moulding, pouring or rolling processes. Household candles are usually made by moulding processes.

Wax candles produced according to the present invention can be dip-coated with a higher melting point wax in order to reduce dripping.

The present invention can be applied to the production of all types of wax candles, including for example church candles, shaped decorative candles, porch light lantern candles and night lights.

The invention is illustrated by the following examples, which should not be regarded as limiting the scope of the invention in any way.

ExamplesPreparation of Composition

Table 1: Elastomeric Polymers.

Polymer	Material Description
KD1102	S-B-S polymer of linear structure with a polystyrene content of about 30% w/w and a medium apparent molecular weight.
KG1650	S-E/B-S polymer of linear structure with a polystyrene content of about 30% w/w and a medium apparent molecular weight.
KG1652	S-E/B-S polymer of linear structure with a polystyrene content of about 30% w/w and a low apparent molecular weight.
KG1657	S-E/B-S di-block containing polymer of linear structure with a polystyrene content of about 15% w/w and a medium apparent molecular weight.
KG1750	(EP) _n polymer of multi-arm structure with a high apparent molecular weight.
KG1780	(S-EP) _n polymer of multi-arm structure with a polystyrene content of 7% w/w and a high apparent molecular weight.
ENGAGE 8200	Saturated ethylene-octene copolymer.
EXACT 4049	Saturated ethylene-butene copolymer.

In the above Table 1, S-B-S refers to a styrene-butadiene-styrene block copolymer, S-E/B-S refers to a styrene-ethylene/butylene-styrene block copolymer, (EP)_n refers to an ethylene/propylene multi-arm copolymer and (S-EP)_n refers to a styrene-ethylene/propylene multi-arm copolymer.

Table 1 details some of the elastomeric polymers that have been tested in wax compositions. For the purposes of Table 1, apparent molecular weights are defined as high,

- 11 -

medium or low, wherein low corresponds to apparent molecular weights of less than 100,000, medium corresponds to apparent molecular weights in the range of from 100,000 to 150,000 and high corresponds to apparent molecular weights of greater than 150,000.

Table 2: Analysis of Scale Waxes.

Characteristic	Wax	
	EXXON SW 130	SHELL SW 250
Congealing Point, °C (ASTM D 938)	51.0	57.2
PEN at 25 °C, 0.1 mm (IP 376/86(95))	48	17.0
Saybolt colour (ASTM D 156)	+24	
Oil Content, %w/w (ASTM D 721)	2.9	4.8
Odour (rating 1-4) (ASTM D 1833)	2.5	

The characteristics of 'EXXON' SW 130 and 'SHELL' SW 250 waxes that were used in testing are detailed in Table 2. The modification of the wax (200 g) was performed in a 400 ml beaker with a high shear rotar/stator mixer equipment (IKA, 'ULTRA-TURAX'). The polymer was dispersed into the wax preheated to 120 °C with a heated oil bath and mixed at 2000 rpm until visibly homogeneous.

Testing

The drop point is the temperature at which the material becomes fluid enough to drop out of a 2.8 mm diameter bottom hole cup. The drop point was measured using a METTLER FP Thermosystem with a temperature sweep of 1 °C per minute.

The disposition of the petroleum waxes to stain was assessed by the following bleeding test, which is similar to that described in *Petroleum Refiner*, 1948, Vol. 27, No. 8, pp. 429-431, referred to above.

- 12 -

Prior to testing the wax under investigation was heated up to 95 °C and then cooled until the sample was 10 °C above the melting point temperature. The bleeding number was measured by allowing a droplet of said liquid wax (diameter 6-8 mm) to fall onto a sheet or a filter paper ('WHATMAN' No. 50, 12.5 cm diameter) and congeal. The sample was placed in an oven at a temperature from 30 to 45 °C for 24 hours. The bleeding number consists of measuring the diameter of the oil ring surrounding the droplet and in subtracting the diameter of the droplet of wax. At least three readings were taken in each case, wherein said differences did not vary by more than 1 mm.

Examples 1-6

Table 3: Effect of Elastomeric Polymers on Scale Wax SW 130 Properties.

Example	Elastomeric Polymer (2 %w added)	Wax SW 130		
		Drop Point Temperature (°C)	Bleeding Number (mm)	
			35 °C	40 °C
Comparative 1	none	50	10	22
2	'KRATON' G 1650	63	0	4
3	'KRATON' G 1652	53	0	0
4	'KRATON' G 1780	52	0	0
5	'KRATON' G 1657	51	0	0
6	'KRATON' G 1750	52	0	0

Table 3 details the results of bleeding number tests and drop point measurements for various 'KRATON' G elastomeric polymer/SW 130 wax compositions. Results are compared with unmodified SW 130 scale wax. The modification of scale waxes containing less than 5% oil, with any 'KRATON' G polymer reduced the oil bleeding tremendously and improved the dry feel of the blends to a level matching that of a fully refined paraffin wax.

- 13 -

In Examples 2, 3, 4, 5 and 6, the addition of 2% w/w of a 'KRATON' G elastomeric polymer resulted in a bleeding number of 0 mm at normal operating temperature (35 °C) compared to unmodified SW 130 scale wax in Example 1 which exhibited a bleeding number of 10 mm under identical conditions.

Examples 7-15

Table 4: Effect of Elastomeric Polymers on Scale Wax SW 250 Properties.

Example	Polymer Type	Polymer Concentration (% w/w)	Drop point test (°C)	Hardness Shore A (0 sec.)	Bleeding Number (mm)			
					30 °C	35 °C	40 °C	45 °C
Comparative 7	pure SW 250	---	58	33	2	6-4	10-7	12-10
8	KD 1102	2			2	4-4		
9	KG 1650	2	69	49	0	0.5	4-4	5-7
10	KG 1652	2	61	52	0	0.5	4-3	4-3
11	KG 1657	2	60	55	0	0	1-2	1-2
12	KG 1657	3	60	56				0
13	'ENGAGE 8200'	2	60		0	1	8-8	8-8
14	'EXACT 4049'	2	60		0	0	4-5	5-6
15	Blend KG 1780 + 'EXACT 4049'	(2+1)						0
16	KG 1780	2	60	49	0	0	0	0

In Table 4, KD and KG mean 'KRATON' D and 'KRATON' G respectively.

Table 4 details the results of bleeding number tests and drop point measurements for various 'KRATON' G elastomeric polymer/SW 250 wax compositions. Results are compared with unmodified SW 250 scale wax.

Where several values appear in the bleeding number results, these indicate a bleeding number analysis that has been carried out on both the horizontal and vertical axes.

The results illustrate the effect of adding a small amount of elastomeric polymer to the wax composition on the bleeding number of said wax. The incorporation of elastomeric polymer generally causes a reduction in bleeding number when compared to the unmodified SW 250 wax compositions in Comparative Example 7.

The incorporation of 2% w/w of 'KRATON' G polymer to the wax composition in Examples 9-11 and 16, was sufficient to reduce bleeding number to 0 mm at 30 °C. Even at elevated temperatures up to and including 45 °C, the incorporation of 2% w/w of these polymers caused at least a 30% reduction in bleeding number as compared to unmodified SW 250 wax.

In Example 12, the incorporation of an additional 1% w/w of elastomeric polymer (to give 3% w/w polymer content) was sufficient to further reduce the bleeding number to 0 mm at 45 °C upon addition of 3% w/w elastomeric polymer.

Examples 13 and 14 indicate the beneficial effect that the addition of a metallocene polymer to the wax composition has on the bleeding number. Both 'ENGAGE 8200' and 'EXACT 4049' polymers result in a bleeding number of 0 mm at 30 °C for the SW 250 wax composition. Metallocene polymers were also found to have

- 16 -

a significant whitening effect on the wax composition produced.

In Example 15, a blend of 'KRATON' G 1780 and metallocene polymer, 'EXACT 4049', 2:1 % w/w, for whiter wax blend resulted in a bleeding number of 0 mm at higher temperatures of 45 °C.

Candles

Candles were prepared from wax blends containing elastomeric polymer and compared to candles made from conventionally de-oiled slack waxes.

Deoiling of 'EXXON' slackwax SW 130

'EXXON' Slackwax SW 130 was deoiled under the following conditions:

Solvent: MEK/toluene 60/40 v/v

Solvent/feed ratio: 3:1 v/w

Wash/feed ratio: 2:1 v/w

Deoiling temperature: +5 °C

Wax recovery: 84.9% w, (Congealing point: 53.3 °C), Oil Content < 1% w/w

Candle Moulding

Candles (diameter 22 mm) were moulded from compositions containing SW 130 wax and 2% w/w of various elastomeric polymers. Similar candles were produced from deoiled 'EXXON' SW 130 slack wax (22 mm diameter).

Candles were moulded in glass pipes of internal diameter (ID) of 22 mm. Hot molten wax (ca. 100 °C) was poured into the ambient temperature glass pipes. A 'WEDO' flat wick, 3x9 yarns was used in all cases. When the wax started to crystallise, the pipes were placed in a water bath at ca. 20 °C for about 1 hour. If the candle formed would not release easily from the glass pipes, the pipes were put in ice for a further period (ca. 1 hour), until the candle could be easily removed.

- 17 -

Candle Burning PropertiesExamples 16-18

5 After burning the tip of the candles, the candles' burning rates were observed for 2 hours. The candle was then extinguished. After a rest period of ca. 1 hour the burning test was continued for a further hour.

Table 5: Candle Burning Tests.

Example	Candle	Diameter (mm)	First 2 hours Burning Time		Next 1 hour Burning Time	
			Ave. Rate (g/hr)	Flame Size (mm)	Ave. Rate (g/hr)	Flame Size (mm)
Comp.16	Wax ex SW 130	22	7.3	45-50	6.8	45-50
17	SW130 + KG1652	22	6.7	ca. 40	melted down	---
18	SW130 + KG1657	22	5.5	30-35	6.7	ca. 35

10 In Table 5, KG means 'KRATON' G. Table 5 compares the burning characteristics of candles made from 'KRATON' G elastomeric polymer/SW 130 wax blends to those of fully de-oiled SW 130 slack wax. Wax blends containing elastomeric polymers as oil retention agents are suitable for burning and give comparable results to fully de-oiled slack waxes.

C L A I M S

1. The present invention provides a method for reducing bleeding number in wax compositions comprising wax and oil, wherein said waxes are selected from petroleum and synthetic waxes and said oil is present in the range of 1 to 45% w/w, by incorporating in the composition an effective amount of an oil retention agent in the form of an elastomeric polymer.

2. A method according to claim 1, wherein the elastomeric polymer is a polymer of at least one conjugated diene or a copolymer of at least one mono vinyl aromatic monomer and at least one conjugated diene or a metallocene polymer.

3. A method according to claim 2, wherein the elastomeric polymer is a block copolymer of styrene with butadiene or isoprene.

4. A method according to claim 3, wherein the elastomeric polymer is a hydrogenated block copolymer.

5. A method according to claim 4, wherein the styrene content of the elastomeric polymer is in the range of from 0 to 50% w/w.

6. A method according to any one of claims 1-5 wherein an amount of elastomeric polymer in the range of from 0.1 to 10% w/w is incorporated in the wax composition.

7. A method according to any one of claims 1-6 wherein an amount of elastomeric polymer in the range of from 0.1 to 5% w/w is incorporated in the wax composition.

8. A method according to any one of claims 1-7, wherein the amount of elastomeric polymer is such that the bleeding number is reduced by at least 30% measured at a temperature 30 °C.

- 19 -

9. A method according to any one of claims 1-8 wherein the wax composition is in the form of a candle.

10. The use of elastomeric polymers for reducing bleeding number in wax compositions.

INTERNATIONAL SEARCH REPORT

Inter: national Application No

PCT/EP 00/02848

A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08L91/08 C08L91/06 C11C5/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 C08L C11C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3 857 805 A (PRICKRIL W) 31 December 1974 (1974-12-31) column 1, line 10-26 column 1, line 51 -column 2, line 55 -----	1,5-7,9, 10
A	US 4 224 204 A (NICHOLS JAMES W ET AL) 23 September 1980 (1980-09-23) column 1, line 54 -column 3, line 44 claim 1 -----	1,6,10
A	US 3 554 950 A (SAUER RICHARD W) 12 January 1971 (1971-01-12) column 2, line 3-41 -----	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

21 June 2000

Date of mailing of the international search report

30/06/2000

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Leroy, A

INTERNATIONAL SEARCH REPORT

...formation on patent family members

International Application No

PCT/EP 00/02848

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 3857805	A	31-12-1974	NONE	
US 4224204	A	23-09-1980	NONE	
US 3554950	A	12-01-1971	NONE	

THIS PAGE BLANK (USPTO)